THERMO PROPERTIES OF QUATERNARY MOLTEN SALT HEAT TRANSFER FLUID FOR HEAT RECOVERY SYSTEM

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# DEDICATION

I dedicate this thesis to my parents, Tukimon Bin Markon and Rosnah Binti Musanib, whose I love the most because of everlasting love and support throughout these years.

To all siblings, thank you for the non-stop support and prayers for me to get through the study.

To my beloved friend and all those who were directly or indirectly helping me on giving info and support for this thesis to be completed.

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# ABSTRACT

In the context of the heat recovery system, the application of heat transfer fluid is one of the essential factors that can improve the efficiency of the system. According to the previous research, quaternary molten nitrate salt mixture has been proved to be an excellent heat transfer fluid in the heat recovery system. The objective of this study is to determine the formulation of quaternary molten nitrate salt mixture that has high heat capacity ($C_p$) with a low melting point. Besides that, the thermal properties and the heat transfer rate of the formulation have been determined through several testing. As for the methodology, the formulation of quaternary molten nitrate salt has been mixed and dried at 100$^o$C for 24 hours to homogenize the mixture. At the beginning of the heating process, the mixture was heated up to 150$^o$C and had been remained constant for four hours. Then, the temperature has risen up to 400$^o$C. The mixing was done as the mixtures were cooled until it reached room temperature. From the result of DSC testing, the melting point for Sample 5 with a composition of 10wt% $LiNO_3$ + 10wt% $NaNO_3$ + 40wt% $KNO_3$ + 40wt% $Ca(NO_3)_2$) has been verified to be below 100$^o$C, which was 97.71$^o$C with the highest heat capacity of 0.46J/g$^o$C. From TGA testing, the liquidus temperature and maximum thermal stability temperature of Sample 5 were detected at the temperature of 36.03$^o$C and 439.04$^o$C respectively. For the heat transfer performance test, during the "Heater on" condition, it is shown that as the range of flow rate increased, the heat transfer rate has increased for all setting temperatures. As for the "Heater off" condition, Sample 5 has lost the least heat to the surrounding compared to other selected samples. In conclusion, Sample 5 with a composition of (10wt% $LiNO_3$ + 10wt% $NaNO_3$ + 40wt% $KNO_3$ + 40wt% $Ca(NO_3)_2$) has been identified as an excellent formulation of quaternary molten nitrate salt that has potential to act as heat transfer fluid in the heat recovery system.

# ABSTRAK

Dalam konteks system pemulihan haba, aplikasi cecair pemindahan haba menjadi satu factor yang penting untuk meningkatkan kadar kecekapan system tersebut. Menurut kajian yang lepas, campuran garam kuaternari jenis nitrat telah dibuktikan menjadi cecair pemindahan haba yang terbaik dalam system pemulihan haba. Objektif kajian ini adalah menentukan formulasi campuran garam jenis kuaternari yang mempunyai kadar kapasiti haba yang tinggi dan takat suhu yang rendah. Selain itu, dengan menggunakan formulasi ini, ciri-ciri termal dan kadar peredaran haba untuk formulasi tersebut telah dikenalpasti melalui beberapa ujikaji. Bagi metodologi, campuran garam nitrat jenis kuaternari telah dicampurkan dan dikeringkan pada suhu 100$^{o}$C selama 24 jam untuk melengkapkan campuran. Proses pemanasan bermula dengan memanaskan campuran pada suhu 150$^{o}$C selama empat jam. Selepas itu, suhu telah dinaikkan sehingga ianya mencecah 400$^{o}$C. Campuran ini terhasil setelah campuran ini dibiarkan sehingga suhu menurun ke suhu bilik. Melalui keputusan daripada ujian DSC menunjukan bahawa, takat lebur Sampel 5 dengan campuran 10wt% $LiNO_3$ + 10wt% $NaNO_3$ + 40wt% $KNO_3$ + 40wt% $Ca(NO_3)_2$) telah dibuktikan berada di bawah 100$^{o}$C, iaitu 97.71$^{o}$C dengan kapasiti haba tertinggi iaitu 0.46J/g$^{o}$C. Melalui ujian TGA, suhu penyejatan dan kestabilan haba bagi Sample 5 telah dikenalpasti pada suhu masing-masing iaitu 36.03$^{o}$C dan 439.04$^{o}$C. Untuk ujian pemindahan haba, semasa ujian "*Heater on*" menunjukkan semakin tinggi kadar aliran, semakin tinggi kadar perpindahan haba untuk setiap ketetapan suhu. Manakala untuk keadaan"*Heater off*", Sampel 5 kehilangan haba yang paling rendah berbanding sampel yang lain. Kesimpulannya, Sampel 5 dengan komposisi 10wt% $LiNO_3$ + 10wt% $NaNO_3$ + 40wt% $KNO_3$ + 40wt% $Ca(NO_3)_2$ telah dikenalpasti sebagai formulasi campuran garam nitrat jenis kuaternari yang mempunyai kebolehan untuk menjadi cecair pemindahan haba bagi sistem pemulihan haba.

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# LIST OF SYMBOLS AND ABBREVIATIONS

| | | |
|---|---|---|
| $AgNO_3$ | - | Silver Nitrate |
| $Ba(NO_3)_2$ | - | Barium Nitrate |
| $BaF_2$ | - | Barium Fluoride |
| $Ca(NO_3)_2$ | - | Calcium Nitrate |
| $CO_2$ | - | Carbon Dioxide |
| CH4 | - | Methane |
| CSP | - | Concentrated Solar Power |
| CsF | - | Cesium Fluoride |
| DTA | - | Differential Thermal Analysis |
| DRORC | | Double stage regenerative ORC |
| DSC | - | Differential Scanning Calorimeter |
| g | - | Gram |
| GHGs | - | Greenhouse Gasses |
| $h$ | - | Enthalpy |
| J | - | Joules |
| HTF | - | Heat Transfer Fluid |
| K | - | Kelvin |
| KF | - | Potassium Fluoride |
| $K_2CO_3$ | - | Potassium Carbonate |
| KCl | - | Potassium Chloride |
| $KNO_3$ | - | Potassium Nitrate |
| kW | - | Kilo Watt |
| kg/s | - | Kilogram Per Second |
| kJ/kg | - | Kilo Joules Per Kilogram |
| $LiNO_3$ | - | Lithium Nitrate |

| | | |
|---|---|---|
| $Li_2CO_3$ | - | Lithium Carbonate |
| LiCl | - | Lithium Chloride |
| LiF | - | Lithium Fluoride |
| $\dot{m}$ | - | Flow Rate |
| mg | - | Milligram |
| min | - | Minute |
| mm | - | Millimeter |
| MW | - | Mega Watt |
| $Mg(NO_3)_2$ | - | Magnesium Nitrate |
| $MgCO_3$ | - | Magnesium Carbonate |
| $MgF_2$ | - | Magnesium Fluoride |
| $Na_2CO_3$ | - | Sodium Carbonate |
| NaCl | - | Sodium Chloride |
| NaF | - | Sodium Fluoride |
| $NHNO_3$ | - | Ammonium Nitrate |
| N/A | - | Not Applicable |
| $NaNO_3$ | - | Sodium Nitrate |
| $NaNO_2$ | - | Sodium Nitrite |
| $NO_3$ | - | Nitrate |
| $NO_2$ | - | Nitrite |
| $O_2$ | - | Oxygen |
| ORC | - | Organic Rankine Cycle |
| PCMs | - | Phase Change Material |
| $\dot{Q}$ | - | Heat Transfer Rate |
| RbF | - | Rubidium Fluoride |
| SRORC | | Single stage regenerative ORC |
| STP | - | Standard Temperature And Pressure |
| $Sr(NO_3)_2$ | - | Strontium Nitrate |
| $T_1$ | - | Inlet Temperature |
| $T_2$ | - | Outlet Temperature |
| TGA | - | Thermo Gravimetric Analysis |
| WHR | - | Waste Heat Recovery |

| | | |
|---|---|---|
| $^{o}C$ | - | Degree Celsius |
| $5^{th}$ | - | Fifth |
| $8^{th}$ | - | Eight |
| % | - | Percent |
| $>$ | - | Greater than |
| $<$ | - | Lower than |
| wt% | - | Weight Percent |

# LIST OF APPENDICES

# LIST OF PUBLICATION

**Journals:**

(i) Tukimon, M. F. et al. (2017) 'Characterization and Thermal Properties of Nitrate Based Molten Salt for Heat Recovery System', Journal of Physics: Conference Series, 914(1). doi: 10.1088/1742-6596/914/1/012016.

(ii) Tukimon, M. F. *et al.* (2017) 'Heat Transfer Performance of Quaternary Molten Nitrate Salt Mixture for Heat Transfer Application', 9(4), pp. 9–14.

(iii) Tukimon, M. F. *et al.* (2019) 'Thermal Analysis of Quaternary Molten Nitrate Salts Mixture for Energy Recovery System', 796, pp. 74–79. doi: 10.4028/www.scientific.net/KEM.796.74.

# CHAPTER 1

# INTRODUCTION

Chapter 1 explains the background of the study, the problem statement and the objectives. Lastly, it is mentioned about the scope and the significance of the study were mentioned.

## 1.1. Research Background

In recent years, the use of molten salt as heat transfer fluid has become the preferable composition among other researchers since it has excellent properties such as low melting point, high heat capacity and has a wide range of temperature in terms of heat recovery system (Ren *et al.*, 2014). From the previous researcher, molten salt is defined as a salt that exists in the solid phase at standard temperature and pressure (STP). Still, due to the increment of temperature, the molten salt existed in the liquid phase. These molten salts can be a great source depending on the application that suitable for it. According to Olivares, molten salt is a substance that can sustain high temperatures before it is fully decomposed (Olivares, 2012). The molten salt composition is both non-toxic and inert liquid. It is frequently used as a heat transfer fluid as well as a thermal energy storage medium in various applications such as concentrated solar power (CSP) and electricity. The characteristic of molten salt itself plays an essential role in the molten salt that can act as high thermal energy storage and heat transfer application. For example, low melting points, moderate density, contain high heat capacity and excellent thermal stability are the most crucial information that needs to be considered in molten salt for these roles to be achieved (Wang *et al.,* 2013). By using molten salt compositions that possess high thermal energy storage capacity has increased the efficiency and enhanced the operation of the system. In order to get a low melting point molten salt mixtures but has high thermal storage for a system, a new quaternary eutectic system consisting of lithium nitrate, sodium nitrate, potassium nitrate and sodium nitrite ($LiNO_3$ - $NaNO_3$ - $KNO_3$ – $NaNO_2$) was developed by the previous researcher. It is stated that the melting point for this eutectic composition was below 100$^{o}$C which was 99.02$^{o}$C (Wang *et al.*, 2013).

Nowadays, molten salts have been used in many industries as a high-temperature heat transfer sources (Redzuan *et al.,* 2016). Molten salts are selected due to their properties such as it is a liquid at atmospheric pressure, a non- flammable, low cost and very efficient in storing energy. The properties of molten salt as heat transfer fluid such as the ability to store the collected energy and deliver it during the time of absence of the source itself was one of the most critical requirements. Based on previous research, it is proved that the efficiency of the Rankine cycle in the steam turbine system has increased by using molten salt as the medium of heat transfer fluid (Kearney *et al.*, 2004). By using molten salts, it is possible to extract and utilize heat energy for useful purposes in the heat recovery system.

Molten salts have many advantages such as high heat capacity and a wide range of operating temperatures. Due to these properties, molten salts can be excellent heat transfer fluid (HTF) in the heat recovery system which has increased the heat storage in the low-temperature heat recovery system. Currently, available molten salt heat transfer fluid based on the salt that has a high melting point and expensive. This high melting point limits the practicality of molten salts as heat transfer fluids in the low-temperature recovery system. As mentioned from previous researchers, it states that available molten salt heat transfer fluid has a high melting point from 140$^{o}$C and above (Redzuan *et al.,* 2016). The uses of nitrate mixture are basically due to their properties such as low melting point, low unit cost, high heat capacity, high thermal stability, negligible vapour pressure and high energy storage density (Bradshaw and Siegel, 2009). The molten salt heat transfer fluid can be classified up to four types of mixture which are primary salt, binary salt, ternary salt and quaternary salt. The quaternary salt was found as the most suitable salt to maintain the effectiveness of extracting and storing the energy since it contains high heat storage capability to ensure stability on the continuous cycle (Li *et al.*, 2016).

Thus, the finding of this research has proved that the mixture of molten salts such as quaternary molten nitrate salt can play an essential role in enhancing the overall properties of heat transfer fluid in the heat recovery system.

## 1.2. Problem Statement

There are various applications of heat recovery systems such as solar thermal, geothermal, biomass, industrial and automotive. These applications have disadvantages such as producing a lot of waste heat. In order to handle this weakness, the waste heat can be reuse, recover or recycle to produce into another useful energy. However, to recover a considerable amount of waste heat, a larger evaporator was needed for the system. Alternatively, by using an appropriate low temperature of heat transfer fluid is considered as an effective way to overcome the weakness of present molten salt in the heat recovery system. The present molten salt is not suitable for low-grade heat recovery since it has a high melting point (typically 240$^{o}$C). In this study, a mixture of molten salt heat transfer fluid based on nitrate salt was proposed to overcome the disadvantages of present molten salt in the heat recovery system. It is expected that a quaternary molten nitrate salt mixture exhibits excellent characteristics of heat transfer fluid such as having a low melting point with high heat capacity and has an excellent heat transfer rate.

## 1.3. Research Objectives

The main objectives of this research are as follows:

(i) To determine the formulation of quaternary molten nitrate salt mixture that gives high heat capacity, $C_p$ with a low melting point.

(ii) To determine the thermal properties of quaternary molten nitrate salt mixture.

(iii) To determine the heat transfer rate of quaternary molten nitrate salts mixture using “Heater on” and “Heater off” conditions through a designated system.

## 1.4. Research Scope and Limitation

The scope of the research consists of three main phases which are:

(i) The formulations were chosen from the thermal properties of eutectic salts made from previous research that consists of lithium nitrate ($LiNO_3$), sodium nitrate ($NaNO_3$), potassium nitrate ($KNO_3$) and calcium nitrate ($Ca(NO_3)_2$).

(ii) The characterization of quaternary molten nitrate salt mixture in order to determine the heat capacity ($C_p$), liquidus and maximum thermal stability temperature has been done by using differential scanning calorimetric (DSC) analysis and thermogravimetric analysis (TGA).

(iii) The heat transfer performance test has been conducted to improve the efficiency of quaternary molten salts on transferring and storing heat energy. The efficiency of heat transfer for each sample was conducted through the “Heater on” and “Heater off” condition. The idea of this “Heater on” condition was to measure the heat transfer rate of samples by using several parameters such as different set temperatures with various ranges of the flow rate of working fluid. The “Heater off” condition was to determine the ability to store heat energy for each sample. From these results, it has improved the ability of molten salt mixtures on transferring and storing heat energy in the heat recovery system.

## 1.5. Significant of Study

In order to maintain the effectiveness of transferring and storing heat energy, it requires a mixture that can store heat to ensure the steadiness of continuous cyclic operation. Molten salts have many advantages such as high heat capacity and a wide range of operating temperatures. Due to these properties, molten salts have been an excellent heat transfer fluid in the heat recovery system which has increased the heat storage within a low-temperature heat recovery system. Currently, available heat transfer fluid based on the molten salt has a high melting point and very expensive in costs. This high melting point limits the practicality of molten salts as heat transfer fluids in the low-temperature heat recovery system. Thus, this study aims to formulate nitrates based on molten salts with a different constituent of salt from the previous researcher. Besides, this study also identified the correlations between the formulations of the quaternary molten nitrate salt mixtures with the thermal properties of the mixture. The heat transfer performance test has been conducted to measure the efficiency of the quaternary molten nitrate salt in the heat recovery system. The expected outcomes are the understanding of the formulation of the quaternary molten nitrate salt mixture from their thermal properties. At the end of this study, the formulation of the quaternary molten nitrate salt mixture that has excellent thermal properties has been proposed as heat transfer fluid in the heat recovery system.

# CHAPTER 2

# LITERATURE REVIEW

The purpose of this chapter is to provide an understanding of quaternary molten nitrate salts mixture. This chapter discusses the usage and the benefit of using quaternary molten nitrate salts as heat transfer fluid. Finally, this chapter has information on previous studies that have been conducted to get new ideas in the application of quaternary molten nitrate salts in the heat recovery system.

## 2.1. Heat Recovery System

Nowadays, the crisis such as increasing global warming has been a concerning topic due to the calamity to our earth. This is because more than half of the energy such as waste heat from the heat is lost to the environment. From the previous researcher has come up with the idea of converting this wasted heat energy into a convenient form like electricity depends on the temperature of the waste heat (Imasato *et al.*, 2019). Industrial waste heat should be recovered and reused to make it eco-friendly in our daily life. Generally, waste heat is split up into three parts according to the temperature which are low grade (<230°C), medium grade (230–650°C) and high grade (>650°C) (Chen *et al.*, 2017; Zhi *et al.*, 2019). As for the industrial sector, the high-grade of waste heat has already been reused. However, waste heat under the temperature of 200°C (Chen *et al.*, 2017) is still occurring in the atmosphere and is not taken seriously by the industry for society. The low-grade waste heat energy was mostly released

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